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INTERACTION BETWEEN PERFLUOROALKYL POLYETHER LINEAR AND CYCLIC DERIVATIVE FLUIDS AND HIGH TEMPERATURE BEARING STEELS IN OXIDATION CORROSION ENVIRONMENT

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FOREWORD

This report was prepared by the Lubricants and Tribology Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with George J. Morris, Project Engineer. Work was initiated under Project No. 7343, "Aerospace Lubricants," Task No. 734303, "Fluid Lubricant Materials." The report covers work accomplished from March 1972 to January 1973. It was submitted by the author in April 1973.

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This technical report has been reviewed and is approved.

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ABSTRACT

This report describes the interaction between perfluoroalkyl polyether linear and cyclic derivative fluids and a high temperature bearing steel, WD-65, among others, studied in a laboratory oxidation-corrosion environment. Temperature ranges of 500°F to 700°F were covered by this investigation. Two hardness levels of WD-65 alloy showed little reactivity with the perfluorinated fluids as well as with dissimilar and ferrous metals up to 700°F. The WD-65 did not catalyze the failure of either dissimilar or ferrous alloys nor did it interfere with additive response in the fluids. It was substantially less reactive than M-50 or 52100 bearing steel alloys in the perfluoroinated fluids of interest.

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INTRODUCTION

The trends of advanced performance aerospace systems toward high temperature operation have led to the development of both metals and fluid lubricant materials over the past decade that are capable of withstanding stringent thermal and oxidative stresses without undergoing appreciable degradation. However, in the gas turbine engine oil area high thermal and oxidative stability must also be accompanied by other specific attributes such as metal compatibility, lubricity, and reasonable rheological properties at low temperatures. This has presented a problem, for example, in the case of the polyphenyl ethers typified by the 5P4E structure. They have outstanding oxidative and thermal stability but are unfortunately accompanied by poor low temperature flow characteristics and poor lubricating capability (without additives) which fall short of meeting gas turbine engine oil needs. Currently used silicones have excellent rheological properties, fairly good lubricating capabilities, but were found to be lacking in moderately high (428°F to 465°F) temperature oxidative environment (Reference 1). Another class of fluids that appeared to provide most of the above-mentioned properties consisted of a series of polymeric perfluorinated polyether fluids that also provided chemical inertness, complete nonflammability, and nonsludging or lacquer formation tendencies. The first of these known as the "Krytox" oils was produced by duPont with varying molecular weight ranges and was recommended for use in applications such as high temperature hydraulic fluids and gas turbine engine oils. Their fluid range was not y acceptable because of a sacrifice of proper fluidity at -20°F to maintain reasonable viscosity

above 1.0 centistoke at 500°F and sufficient volatility at 400°F. It was also demonstrated by former workers in this laboratory that these fluids could be inhibited with additives (solubility sometimes difficult) to increase their useful range at high temperatures by approximately 100°F (Reference 2). A similar fluid was also commerically produced by another manufacturer at a later date. This technology was further extended by duPont by synthesizing the perfluoroalkylpolyether with one or more triazine structures included. The fluids described above were evaluated with metals of interest under oxidation corrosion conditions from 500°F up to 700°F.

In the metals area the Metals and Processing Branch of the Metals and Ceramics Division, Air Force Materials Laboratory, efforts were directed toward developing bearing materials with superior elevated temperature properties over 900°F. One of the most commonly used bearing steels, 52100, could no longer be considered in this high temperature regime (1000°F) because of a rapid loss of hardness and dimensional stability. Hot worked die steels and high speed tool steels met the requirements for aircraft bearings with only varying degrees of success. As a result of the work carried out by Crucible Steel under AFML contract (Reference 3) a prototype high temperature alloy designated WB-49 was developed for aircraft bearing application over 900°F. This technology was extended to incorporate corrosion resistance to the already attained high initial hardness, adequate temper resistance (in the range of 600°F and 900°F), and good dimensional stability of the alloy. This composition designated as WD-65 was recommended for use as a corrosion resistant bearing steel for 500 hours of operation at temperatures up to 900°F (Reference 4).

Since the perfluorinated fluids had been shown to have outstanding thermal and chemical stability at high temperatures, their interaction with high temperature metals that they might encounter was of immediate interest. Several concurrent studies were carried out when the fluids were first introduced with emphasis on oxidation stability and metal corrosion tendency One of these was performed by duPont in a semi micro oxidation-corrosion test rig, the details of which appear in Reference 5. This study showed that the perfluoroalkylpolyether fluid, then identified as PR-143 could be used without deleterious effects up to 550°F on most 400 series stainless steels, a high speed tool steel and the bearing steels such as M-1, M-50, 52100 and WB-49. It was also noted that above 600°F, several of the frequently used titanium alloys caused severe degradation to the PR-143 fluid even in the absence of oxygen. An unexpected improvement up to 650°F in corrosion behavior of the bearing steels M-1, M-50, and WB-49 was observed when these materials were hardened and configured for use in ball bearing hardware instead of the washer shaped metal specimens normally used in current oxidation-corrosion testing.

Laboratory investigations conducted by this laboratory on the perfluoroalkylpolyether fluid utilizing slightly different oxidizing conditions showed a considerable corrosive effect on most ferrous metals and certain titanium alloys at 550°F and above. Included in these ferrous alloys were M-1, M-10, 52100, and 440 C that were included in the previously described program (Reference 6).

A more recent evaluation of the interaction between M-50 and WD-65 aircraft bearing steels and perfluoroalkylpolyether gas turbine engine oil grade by this time identified as Krytox 143 AC was performed by the Air Force Aero Propulsion Laboratory (Reference 7). The conditions were again of the oxidation-corrosion variety conducted in accord with the Coordinating Research Council (CRC) procedure L-53-368 at 644°F for 48 hours. This test method was the forerunner of the current Method 5307 for Federal Test Method Standard 791b used to evaluate the oxidation stability of candidate gas turbine engine oils directed toward the requirements of specification MIL-L-27502 (Reference 8). Both metals suffered excessive corrosion by our current standards with the M-50 appearing worse than the WD-65 with the naked eye and under the microscope. The M-50 showed a coated, very rough and pitted surface while the WD-65 was only coated and not as rough (Table I). Based on previcus experience within this laboratory and that of outside workers reviewed herein these results could be expected at the test temperature that was selected. However since these high temperature alloys were considered to be the aircraft bearing steels of the future and similarly the perfluorinated fluids were considered to be the future high temperature class of lubricants, the question arose as to the mutual compatibility of these materials under severe projected use conditions. In order to answer this compatibility question with a relative degree of certainty, a study was conceived utilizing the micro oxidation-corrosion test (Reference 9). This method which has previously been used in high temperature fluid studies, incorporates the severity and repeatability necessary to reliably project the interaction of both metals and perfluorinated fluids at high temperatures. The data accumulated in this study are reported herein.

TABLE I

TEST RESULTS FROM A REFLUX OXIDATION-CORROSION TEST

48 HOURS @ 644°F

PROCEDURE: CRC DESIGNATION L-53-368 (FTMS 791b, METHOD 5309)

METAL SPECIMEN DATA:	Weight Change mg/cm^2	DESCRIPTION
WD-65:		
Before cleaning	+0.96	Dark brown and black coating
After electrocleaning	-1.54	Rough
M-50		
Before cleaning	+1.42	Dark brown and black coating
After electrocleaning	+0.72	Very rough and pitted

NOTE: Pretest appearance both specimens:
Bright, shiny and polished finish
Approx: 5 μ in. rms

SECTION II

OXIDATION-CORROSION CHARACTERIZATION

Oxidation-corrosion (o-c) studies were performed under two basic conditions with the perfluorinated fluids identified below:

Sample Identity	Fluid Identity
MLO-73-20	Perfluoroalkylpolyether, from manufacturer Nr 1
MLO-73-21	Inhibited perfluoroalkylpolyether
MLO-73-22	Perfluoroalkylpolyether (HFPO) triazine
MLO-73-23	Perfluoroalkylpolyether from manufacturer Nr 2

These fluids were furnished as laboratory samples from interested suppliers and are not necessarily typical of fluids that would be supplied on a full scale production. The assumption is made that refinements and improvements on the finished product would be incorporated in scaling up from the laboratory bench to larger scale manufacturing.

All investigations were made in micro scale apparatus which is described in great detail (Reference 9). When dissimilar metal specimens were employed, the glassware configuration contained a condenser for refluxing fluid vapors. When ferrous metals were tested, the condenser was replaced with a side arm adapter in an "overboard" configuration which allowed for collecting and measurement of condensed fluid vapors. All metal specimens were separated by glass spacers as illustrated in Figure 1.

The metal specimens were selected from combinations previously evaluated in perfluoralkylpolyether fluid (their compositions where possible

are covered by federal, military or industrial specifications as listed on Table II). The term "dissimilar" metals refers to a specific metal combination that includes two ferrous metals. The washer type specimens of WD-65 alloy were furnished for this evaluation by the Metals and Processing Branch (LLM) of the Metals and Ceramics Division of AFML. They are identified below:

Identity	Source
WD-65	Colt Industries, Crucible Inc., - fully hardened at heat treatment to Rockwell C "65.5"
WD-65-53	Federal Mogul - did not fully harden at heat treatment - Rockwell C "53"

Washer-shaped specimens of the material from both sources were heat treated as follows:

Preheat at 1550°F/5 min.

Austenitize at 2190 F/2 min; quench into salt at 1000/5 min; air cool to room temperature

Refrigerate at -100 F/30 min

Triple temper at 1000 F/2hr with refrigeration step (-100 F/30 min) between each tempering step.

The final hardnesses were Rockwell "C" 65.5 for the Colt Industries, Crucible Inc., material and Rockwell "C" 53 for the Federal Mogul material. The reasons for the Federal Mogul material not responding to the above heat treatment cycle have not been explored.

The WD-65 and WD-65-53 specimens were investigated alone in each fluid and inserted into each combination of dissimilar and ferrous metals,

TABLE II

METAL TEST SPECIMEN IDENTITIES

Test Specimen Identity	Specification
Dissimilar	
Titanium, 6 Al - 4 V	MIL-T-9046, titanium and titanium alloy sheet, strip and plate, type III, composition C
Aluminum, 2024	QQ-A-250/4, aluminum alloy 2024, plate and sheet
Tool Steel, M-10	AISI, Type M-10, machined from bar
Silver	MIL-S-13282, electrolytic, 99.9, Grade A
301 Corrosion Resistant	MIL-S-5059, Type 301, Half Hard
Ferrous	
4140 steel alloy	Steel alloy, grade 4140 machined from annealed bar
52100	MIL-S-7430B, Grade E, from annealed strip
410	Stainless steel, Type 410 from annealed sheet
M-50, Tool Steel	AMS 6490A, Grade M-50 from annealed sheet
440C	QQ-S-763, Type 440-C machined from annealed bar

first to determine single metal activity and second to determine the degree of interaction of both WD-65's with the metal combinations normally used in oxidation-corrosion studies and the fluids in question.

The failure criteria chosen for this study were essentially the same as those used in previous perfluoroalkylpolyether fluid studies. Attack on the metal specimens (etiher weight gain by deposition or weight loss by corrosion) was considered excessive if the observed change was greater than 0.2 milligrams per square centimeter of specimen area over the original weight. Fluid deterioration criteria were derived from the limits set for long-term gas turbine engine oil goals as shown below:

Total acid number	$\pm$4.0 max
Viscosity change @ 100°F, %	$\pm$25.0 max

No oxidation-corrosion test was carried beyond 700°F since this is beyond the projected limit for the perfluoroalkylether fluids due to viscosity and volatility characteristics.

SECTION III

DISCUSSION OF DATA

Oxidation-corrosion data obtained with the perfluoroalkylpolyether, MLO-73-20, and the various metals are summarized on Table III. The reactivity of WD-65-53 by itself was nil until 650°F was reached. At this temperature attacks on all the specimens was excessive with deposition ranging from heavy black to a heavy multi-colored (peacock) appearance. Degradation of the fluid was minimal at all temperatures which was the rule rather than the exception with other fluids. Fluid condition, therefore, will be considered within the prescribed limits unless otherwise stated throughout the remainder of this report. The WD-65 by itself demonstrated erratic behavior at 550°F but through 575°F and 600°F appeared nonreactive before it finally failed completely at 650°F (showing heavy black deposits). This erratic behavior had been observed at the lower temperature by other investigators.

The WD-65-53 with dissimilar metals in the MLO-73-20 fluid did not show corrosive attack at 500°F. Corrosion began to develop at 550°F along with aluminum and titanium. All dissimilar metals and WD-65-53 showed excessive corrosion at 600°F with the WD-65-53 appearing with a tan tarnish and spotted. A similar behavior pattern was observed with WD-65 and the dissimilar metals.

In oxidation-corrosion characterization with ferrous metals, each hardness level of WD-65 showed no corrosion at 500°F along with the other alloys in MIL-73-20 fluid. This trend of nonreactivity of WD-65's

continued to 600°F despite heavy corrosive attack on all other ferrous alloys. It is apparent that none of the other ferrous metal's failures could be attributed to the presence of either hardness level of WD-65 in this characterization or in others involving the different types of fluids in this study.

Oxidation-corrosion data with the perfluoroalkylpolyether base fluid formulated with a high temperature corrosion inhibitor additive C, identified as MLO-73-21 appears on Table IV. Initial work was begun at a higher temperature level of 650°F with WD-65-53 and WD-65 alone to determine the inhibitor effectivity. No metal attack was observed at 650°F or 700°F with the washers having a slightly stained appearance. Fluid deterioration at 650°F only was indicated for both hardness levels by a gross viscosity loss.

Dissimilar metals with WD-65-53 showed no reactivity with the inhibited fluid MLO-73-21 through 550°F and 600°F and slight reactivity at 700°F with aluminum and M-10. This same trend occurred with WD-65 through 700°F where no reactivity was observed even at that top temperature. All WD-65 specimens at the end of each test were slightly stained in appearance. Fluid deterioration was excessive with the WD-65-53 at 550°F and 700°F as indicated by high viscosity and high volatility losses. By comparison, the WD-65 fluid deterioration at 550°F and 700°F although not excessive were an order of magnitude or two beyond normal expectations.

Reactivity of the inhibited perfluoroalkylpolyether MLO-73-21 with WD-65-53 and ferrous metals was mixed. At 550°F M-50 was excessively corroded and at 600, the 4140 showed attack. Corrosion was excessive on

all the ferrous metals at 700°F. With WD-65 the ferrous metal combination showed very little susceptibility to corrosion at 550 and 600°F. However, at 700°F high corrosion was observed. Interestingly through the above temperature range to 700°F both hardness levels of WD-65 with both metal combinations were not attacked excessively by the inhibited fluid.

Oxidation-corrosion data on the perfluoroalkylpolyether (HFPO) triazine MIL-73-22 is summarized in Table V. Each hardness level of WD-65 when immersed in the above fluid showed no attack at 650°F. At 700°F the WD-65-53 remains nonreactive while the WD-65 at 700°F gave incipient corrosion. Both sets of specimens ranged from a light stain to spot deposits in appearance. With dissimilar metals the WD-65-53 and WD-65 showed no corrosion through 600°F and up to 700°F with the neat fluid and a formulation using the same oxidation inhibitor (additive C) previously used with the perfluoroalkylpolyether. At 700°F the dissimilar metal combination showed excessive corrosion of the titanium and M-10 in duplicate tests with the neat fluid and the inhibited fluid. In addition the silver showed excessive weight change in the inhibited fluid only. Both WD-65's were characterized by a golden stain.

The study of the ferrous metal combination, WD-65's and HFPO triazine also incorporated an additive effectiveness evaluation in addition to the neat fluid reactivity. At 500°F all ferrous metals and WD-65's were not corroded excessively with the neat fluid but at 600°F most of the ferrous were attacked including M-50. Both WD-65's did not suffer excessive weight change. The addition of additive C to the fluid eliminated the corrosion at 600°F on the ferrous combination but was not effective at

650°F. At both of the above temperatures neither WD-65's showed signs of corrosion. The other additives M-4 and additive B were not as effective as additive C in their recommended concentrations in the HFPO triazine at 600°F. These additives are identified on Table VI with their corresponding structures.

Oxidation-corrosion data with the perfluoroalkylpolyether fluid from manufacturer Number 2, MIL-73-23 appears on Table VII. Due to insufficient sample complete characterization of the fluid with all metal combinations and all temperatures of interest was not conducted. Some inferences are made by relating the data obtained on this fluid to that obtained on the similar fluid produced by the other manufacturer.

The reactivity of WD-65 by itself was minimal at 500°F up to 600°F. This same performance could be attributed to WD-65-53 at those temperatures by extending the data accumulated on the perfluoroalkylpolyether of different manufacturer. Both hardness levels showed excessive attack at 650°F with a dark tarnish appearance. Interpolated data in Figure 2 shows failure would occur in the neighborhood of 630°F.

In combination with the dissimilar metals, both WD-65's remained passive from 500°F to 600°F with only the silver specimen exceeding the acceptable corrosion limits. This same behavior of nonreactivity of WD-65's was observed with the ferrous metal group at both of the above temperatures. At 600°F, however, all the other ferrous metals showed excessive corrosion in the presence of WD-65-53. Again utilizing previous data obtained on the similar fluid, corrosion of the ferrous

metals in the presence of WD-65 could be assumed at 600°F. The appearance of the WD-65's throughout the dissimilar and ferrous metal evaluations was dull metallic with no discoloration. Fluid deterioration at 600°F with both mixed metal combinations ranged from borderline passing to failure as indicated by high viscosity changes, volatilities, and acid numbers.

SECTION IV

CONCLUSIONS

As a result of this program the conclusions that were reached are as follows:

1. Both hardness levels of WD-65 showed little reactivity with the currently available perfluorinated fluids up to 700°F.

2. Both WD-65's were nonreactive in the presence of dissimilar metals up to 700°F.

3. Both WD-65's were nonreactive in the presence of ferrous metals up to 700°F.

4. Neither hardness level of WD-65 appeared to catalyze the failure of either dissimilar or ferrous metals.

5. Neither WD-65 contributed to any lack of additive response in the perfluorinated fluids.

6. Both WD-65's were substantially less reactive under all conditions and with all fluids than M-50 or 52100 bearing steels (Figures 3 and 4).

7. The difference in hardness level of R_c 12 between the two WD-65 alloys did not effect their reactivity in the perfluorinated fluids investigated up to 700°F.

TABLE III

WD-65 ALLOY EVALUATION IN PERFLUORINATED FLUIDS, MLO-73-20, OXIDATION-CORROSION STUDIES RESULTS

Fluid Identity	Test Temp. °F	Viscosity Change @ 100°F %	Acid No. Mg KOH/g	Fluid Loss %	Metal Weight Change, Mg/cm^2						Remarks: WD-65 Metal Appearance
							WD 65	R_c 53			
Perfluoro-	550	+3.1	NIL	2.0	+0.03	+0.23	+0.05	+0.09	+0.02		Light tan stain; some peacock, no pitting
alkylpoly-	575	+3.8	NIL	4.0	+0.03	+0.02	+0.02	0.00	+0.01		Light deposit; peacock coloration
ether	600	+2.8	NIL	4.0	+0.01	+0.06	+0.09	+0.10	+0.12		Slight peacock
MLO-73-20	650	+2.8	NIL	4.4	+0.47	+0.31	+0.42	+0.30	+0.24		Heavy black deposit; heavy peacock
							WD-65	R_c 65			
	550	+3.1	NIL	1.0	+0.52	+0.12	+0.07	+1.1	+0.1[illegible]		Light tan stain; some peacock
	575	+3.8	NIL	4.0	-0.01	-0.02	-0.02	-0.07	-0.05		Light deposit; peacock
	600	+3.8	NIL	4.0	+0.08	+0.08	+0.08	+0.05	+0.0[illegible]		Slight peacock
	650	+2.7	NIL	4.7	+0.34	+0.33	+0.44	+0.50	+0.70		Heavy black deposit
					Dissimilar Metals w/WD-65						
					Ti	Al	R_c 53	M-10	Ag	301 St.St.	
	500	+30.6	NIL	15.3	0.00	-0.04	-	-0.02	-0.06	-0.04	None
	500	+11.0	NIL	4.0	+0.04	+0.02	+0.03	0.00	-0.07	[illegible].00	Tan tarnish
	550	+ 3.5	NIL	9.8	+0.36	+0.40	+0.33	+0.14	+0.04	+0.18	Tan tarnish
	600	+ 4.8	NIL	9.8	+0.35	+0.38	+0.52	+0.56	+0.47	+0.57	Tan tarnish, spotted
							R_c65				
	500	+7.2	NIL	2.4	+0.04	+0.02	+0.05	0.00	-0.0[illegible]	+0.04	Tan tarnish
	550	+3.5	NIL	10.0	+0.44	+0.28	+0.47	+0.13	+0.14	+0.12	Tan tarnish
	600	+5.5	NIL	10.0	+0.30	+0.23	+0.47	+0.52	+0.12	+0.25	Tan tarnish spots
					Ferrous Metals w/WD 65						
					4140	52100	R_c 53	410	M-50	440c	
	500	+2.4	NIL	0.5	+0.05	+0.02	+0.03	+0.03	+0.09	+0.02	Light tarnish
	600	+1.7	3.1	3.9	-6.90	+2.48	-0.04	-5.79	-8.27	-3.47	Light black tarnish
							R_c 65				
	500	+1.7	NIL	0.3	+0.05	+0.02	+0.05	+0.02	+0.03	-0.02	Light tarnish
	[illegible]00	+1.7	3.1	6.1	-6.80	+1.51	+0.02	-0.45	-1.88	+2.38	Light black tarnish

TABLE IV

WD-65 ALLOY EVALUATION IN PERFLUORINATED FLUIDS, MLO-73-21, OXIDATION-CORROSION STUDIES RESULTS

Fluid Identity	Test Temp. °F	Viscosity Change @ 100°F %	Acid No. Mg KOH/g	Fluid Loss %	Metal Weight Change, mg/cm²						Remarks: WD-65 Metal Appearance
								WD 65	R_c 53		
Perfluoro-	650	-89.5	0.06	5.5	-0.12	+0.10	+0.05	+0.060	-0.09		Slight stain
alkylpoly-	700	+ 5.6	-	8.4	+0.03	+0.03	+0.01	+0.03	0.00		Slight stain
ether plus							WD 65	R_c 65			
proprietary	650	-89.4	0.22	5.8	+0.05	+0.07	+0.04	+0.08	+0.06		Slight stain
inhibitor	700	+ 5.6	-	8.4	+0.06	+0.07	+0.06	+0.04	+0.01		Slight stain
additive											
MLO-73-21					Dissimilar Metal W/WD 65						
					Ti	Al	R_c 53	M-10	Ag	301 St.St.	
	550	+30.8	-	24.4	+0.02	0.00	+0.02	+0.03	+0.06	+0.01	Slight stain
	600	+11.2	-	6.9	-0.01	+0.02	0.00	-0.07	-0.02	-0.05	Slight stain
	700	-27.4	0.27	29.9	+0.09	+0.23	+0.08	-0.28	-0.17	+0.03	Slight stain with carbon spots
							R_c 65				
	550	+17.6	-	15.3	0.00	-0.02	+0.01	+0.02	+0.02	+0.27	Slight stain
	600	+10.9	-	6.1	-0.02	+0.03	-0.02	-0.04	-0.05	-0.03	Slight stain
	700	+17.2	-	16.6	-0.01	+0.07	+0.09	+0.03	-0.03	±0.09	Slight stain
					Ferrous Metals W/WD 65						
					4140	52100	R_c 53	410	M-50	440c	
	550	+ 1.9	-	0.03	+0.15	+0.15	+0.09	+0.08	+1.10	-0.03	Slight stain
	600	+ 2.6	-	0.6	+0.23	+0.15	-0.01	-0.04	+0.16	0.00	Slight stain
	700	+ 6.0	0.52	26.0	-7.14	+2.5	-0.14	-3.2	-9.63	-14.5	Slight stain with carbon spots
							R_c 65				
	550	+ 2.3	-	0.0	+0.16	+0.08	+0.02	+0.04	+0.06	+0.07	Slight stain
	600	+ 2.2	-	0.3	+0.18	+0.13	+0.04	0.00	+0.13	+0.07	Slight stain
	700	+ 4.1	-	25.0	-5.94	+1.62	-0.20	-7.88	-8.54	-15.4	Heavy mottled deposit

TABLE V

WD-65 ALLOY EVALUATION IN PERFLUORINATED FLUIDS, MLO-73-22,

OXIDATION-CORROSION STUDIES RESULTS

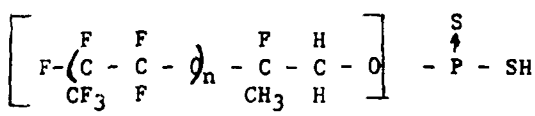
Fluid Identity	Test Temp. °F	Viscosity Change @ 100°F %	Acid No. Mg KOH/g	Fluid Loss %	Metal Weight Change, mg/cm^2						Remarks: WD-65 Metal Appearance
							R_c 53				
HFPO	650	+ 0.23	-	4.81	+0.06	+0.05	+0.04	+0.06	+0.04		No stain or deposits
Triazine	700	- 0.8	-	15.3	+0.09	+0.06	+0.05	+0.09	+0.08		Light stain to spot deposits
MLO-73-22							R_c 65				
	650	+ 0.63	-	4.52	0.00	+0.02	-0.03	-0.03	-0.03		No stain or deposits
	700	+ 0.7	-	16.8	+0.27	+0.22	+0.14	+0.20	+0.17		Light stain to spot deposits
					Dissimilar Metals w/WD-65						
					Ti	Al	R_c 53	M-10	Ag	301 St.St.	
	600	+11.2	-	6.86	-0.01	+0.02	0.00	-0.07	-0.02	-0.05	No stains or deposits
	650	+ 7.0	-	9.1	+0.10	+0.14	-	+0.10	-0.06	+0.05	-
	650	+12.6	-	7.9	+0.05	+0.14	-	+0.03	-0.18	+0.03	-
	700	+25.5	<0.1	26.7	+0.47	+0.14	+0.06	-0.72	-0.05	+0.04	Golden stain
+5% ADD C	700	+12.3	<0.11	21.1	+0.44	+0.10	0.00	-0.80	-0.45	+0.04	Not recorded
							R_c 65				
	600	+10.9	-	6.08	-0.02	+0.03	-0.02	-0.04	-0.05	-0.03	No stains or deposits
	700	+21.4	<0.1	16.9	+0.30	+0.12	+0.13	-0.46	-0.040	0.00	Golden stain
+5% ADD C	700	+ 6.2	<0.12	25.8	+1.48	+0.17	-0.01	+0.56	-0.54	+0.02	Not recorded
					Ferrous Metals w/WD-65						
					4140	52100	R_c 53	410	M-50	440c	
	500	+ 1.8	<0.1	0.6	+0.05	+0.04	+0.03	+0.09	+0.11	+0.08	Tan tarnish
	600	+ 2.7	<0.1	0.3	+0.56	+0.33	+0.16	+0.14	+1.16	+0.17	Blue to tan stain
+2% M-4	600	0.0	<0.1	2.7	+1.4	+1.1	+0.89	+1.1	+1.5	+0.72	Black spotted deposit
+5% M-4	600	0.0	<0.1	5.5	+1.8	+1.7	+2.9	+2.5	+2.2	+3.0	Heavy dark brown to black deposit
+2% ADD B	600	0.9	<0.1	2.4	+0.11	+0.12	+0.06	+0.07	+0.34	+0.06	No stain or deposit
+5% ADD C	600	+ 8.1	<0.1	0.5	+0.10	+0.02	+0.06	+0.07	+0.18	+0.10	No stain or deposit
+5% ADD C	650	+ 3.4	<0.1	0.3	+0.28	+0.19	+0.07	+0.19	+0.28	+0.50	Blue to tan stain
							R_c 65				
	500	+ 1.8	-	0.3	+0.06	+0.04	+0.08	+0.05	+0.11	+0.16	Tan tarnish
	600	+ 1.8	-	0.3	+0.24	+0.15	+0.04	+0.09	+0.58	+0.06	Blue to tan
+2% M-4	600	+ 0.9	-	2.7	+1.5	+1.2	+1.1	+0.77	+1.1	+1.2	Black spotted deposit
+5% M-4	600	- 0.9	-	5.3	+2.6	+2.6	+2.0	+2.1	+2.9	+2.8	Heavy dark brown to black deposit
+2% ADD B	600	+ 0.9	-	2.1	+0.11	+0.08	+0.18	+0.07	+0.25	+0.11	No stain or deposit
+5% ADD C	600	+ 7.2	-	0.5	+0.07	+0.06	+0.08	+0.12	+0.14	+0.11	No stain or deposit
+5% ADD C	650	-12.7	-	0.4	+0.18	+0.19	0.10	-0.12	+0.23	+0.28	Blue to tan stain

TABLE VI

ADDITIVE IDENTIFICATION AND CHEMICAL STRUCTURES

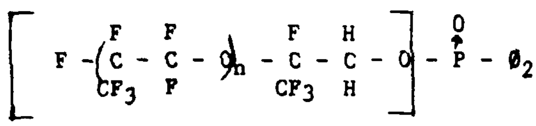
MANUFACTURER: DUPONT

ADDITIVE B - Thiophosphate



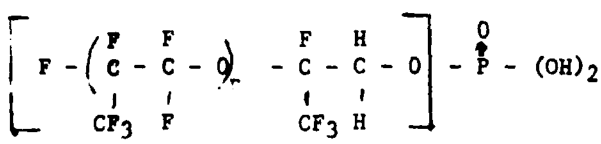
n = 12

ADDITIVE C - Phosphinate



n = ∿ 21

ADDITIVE M-4 - Mono-phosphate



n = ∿ 6

TABLE VII

WD-65 ALLOY EVALUATION IN PERFLUORINATED FLUIDS, MLO-73-23, OXIDATION-CORROSION STUDIES RESULTS

Fluid Identity	Test Temp. °F	Viscosity Change @ 100°F %	Acid No. Mg KOH/g	Fluid Loss %	Metal Weight Change, Mg/cm^2						Remarks: WD 65 Metal Appearance
Perfluoro-alkylpoly-ether, Mfg No. 2 MLO-73-23	500	+ 6.7	NIL	2.5	No Metals						
	600	+405	not sufficient sample	80	No metals						
							R_c 53				
	650	-17.0	-	16.5	+0.25	+0.24	+0.38	+0.31	+0.30		Dark Tarnish
							WD 65	R_c 65			
	500	- 1.32	NIL	1.69	-0.02	0.00	+0.01	0.00	+0.02		
	600	- 1.08	NIL	1.96	-0.01	-0.02	-0.03	-0.02	-0.01		
	650	-11.2	-	10.7	+0.36	+0.41	+0.39	+0.24	+0.22		Dark tarnish
					Dissimilar Metals w/WD 65, R_c 53						
					Ti	Al	R_c 53	M-10	Ag	301 St.St.	
	500	+ 6.81	0.3	1.93	0.00	-0.03	-0.01	0.00	-0.01	-0.01	Dull; no discoloration
	600	-71.3	5.3	31.6	+0.01	-0.04	-0.11	-0.05	-0.45	-0.07	
							R_c 65				
	500	+ 4.25	0.2	0.55	-0.01	-0.03	-0.01	0.01	0.00	0.00	Dull; no discoloration
	600	-23.6	0.61	25.8	+0.02	+0.12	-0.09	+0.08	-0.39	+0.050	
					Ferrous Metals						
					4140	52100	R_c 53	410	M-50	440c	
	500	+ 2.76	NIL	0.0	+0.01	0.00	-0.03	-0.04	+0.01	+0.03	Dull; no discoloration
	600	-82.9	6.6	48.6	-2.27	+0.68	+0.04	-4.07	-4.44	-5.34	
							R_c 65				
	500	+ 3.0	NIL	0.0	+0.05	+0.01	+0.02	0.00	+0.02	+0.02	Dull; no discoloration

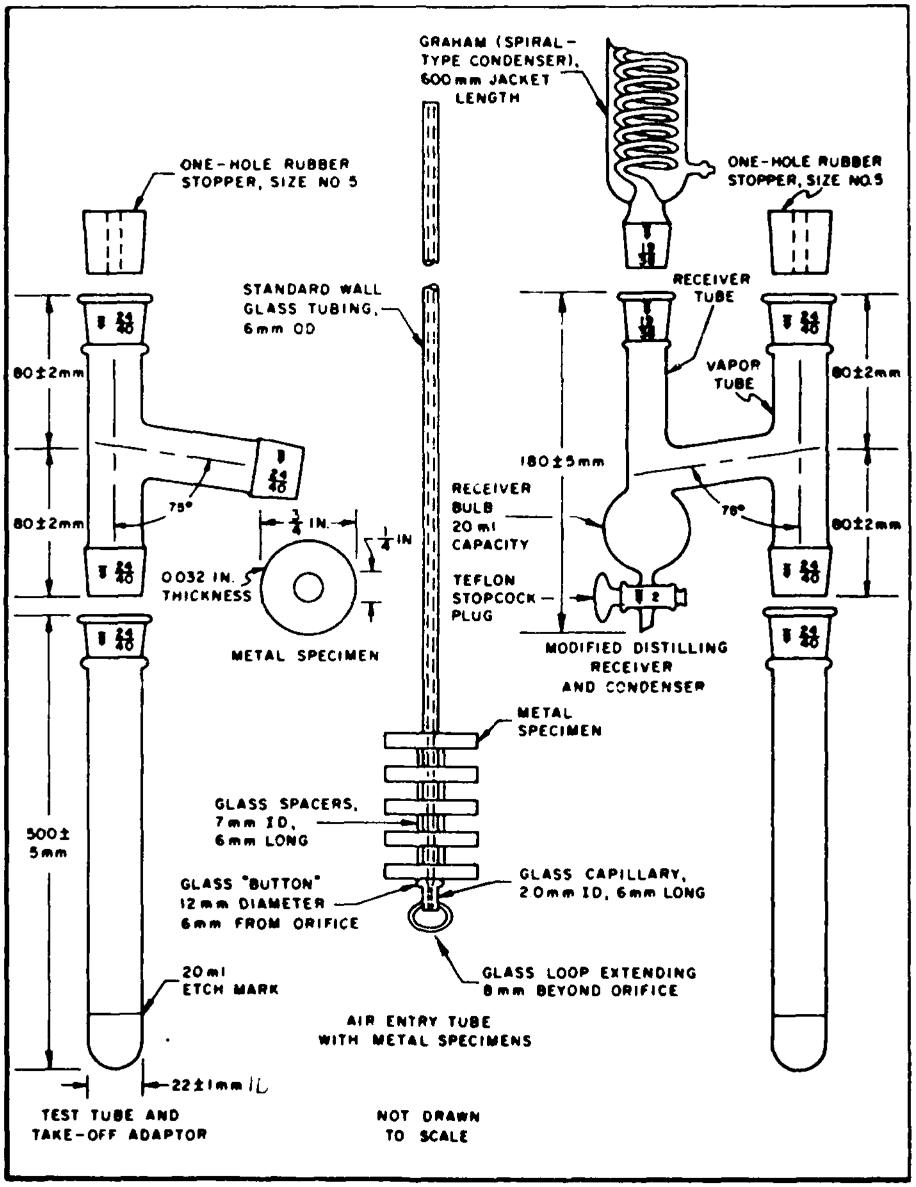


Figure 1. Micro-O-C Test Apparatus

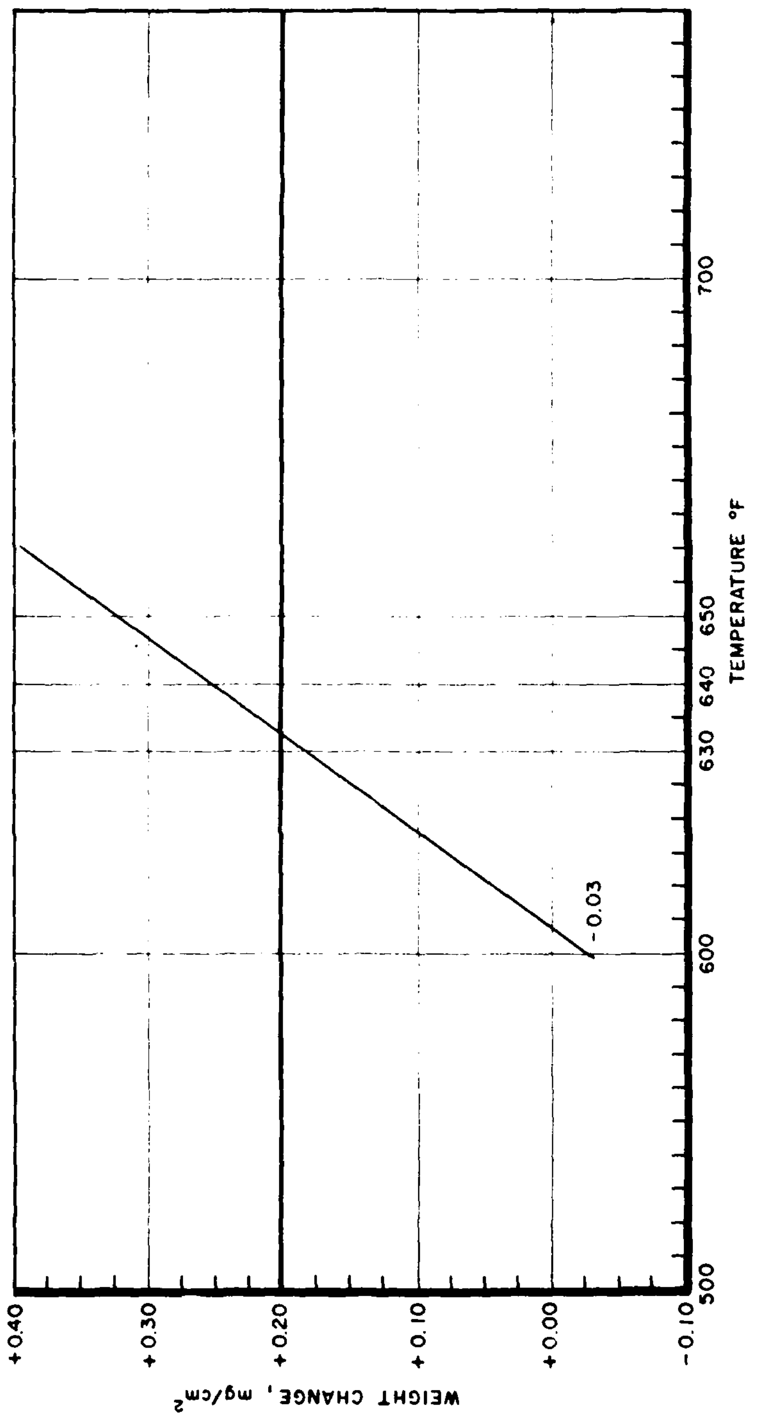


Figure 2. WD-65 Corrosion in MLO-73-23 Fluid

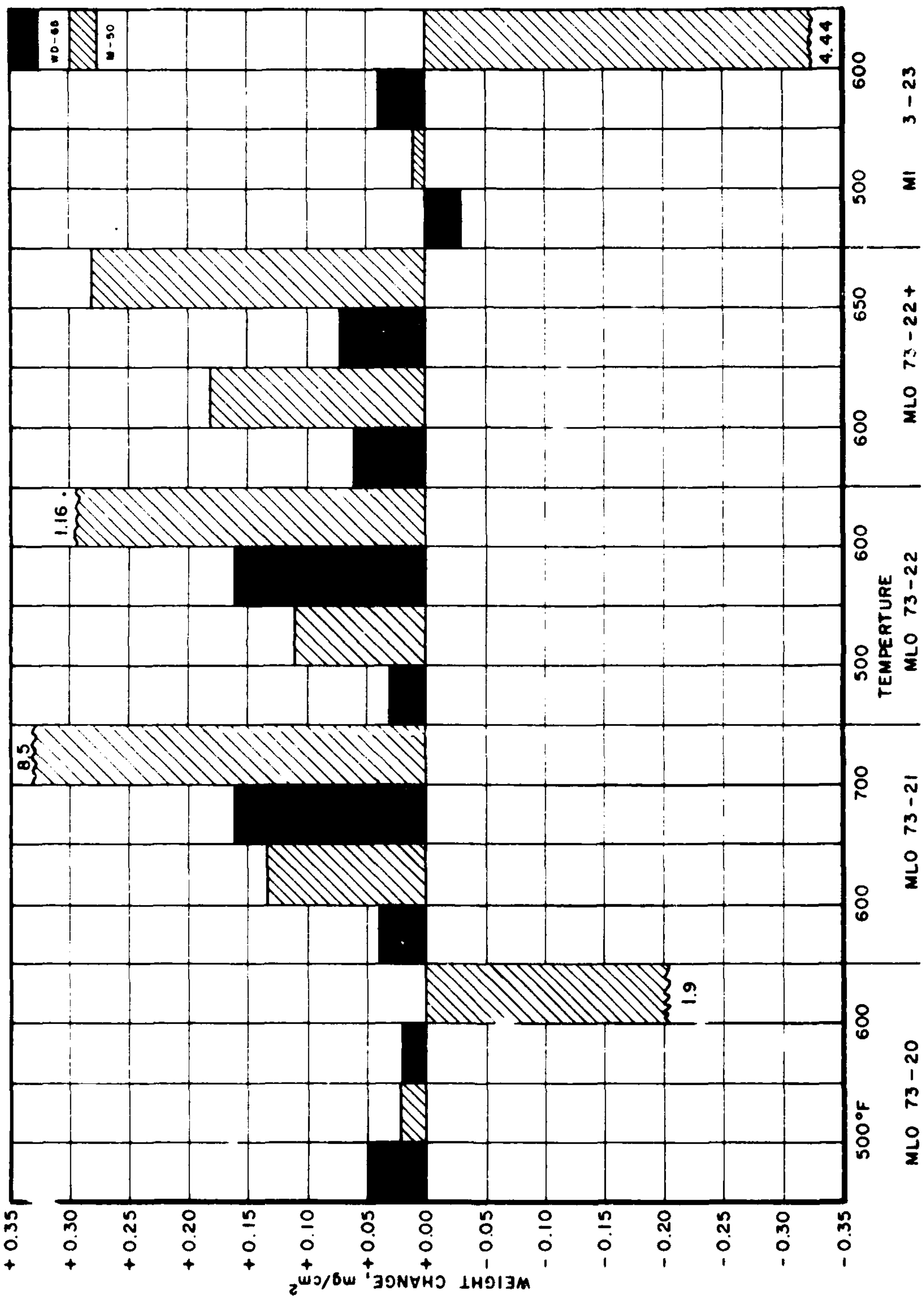


Figure 3. WD-65-M-50 Corrosion Comparison

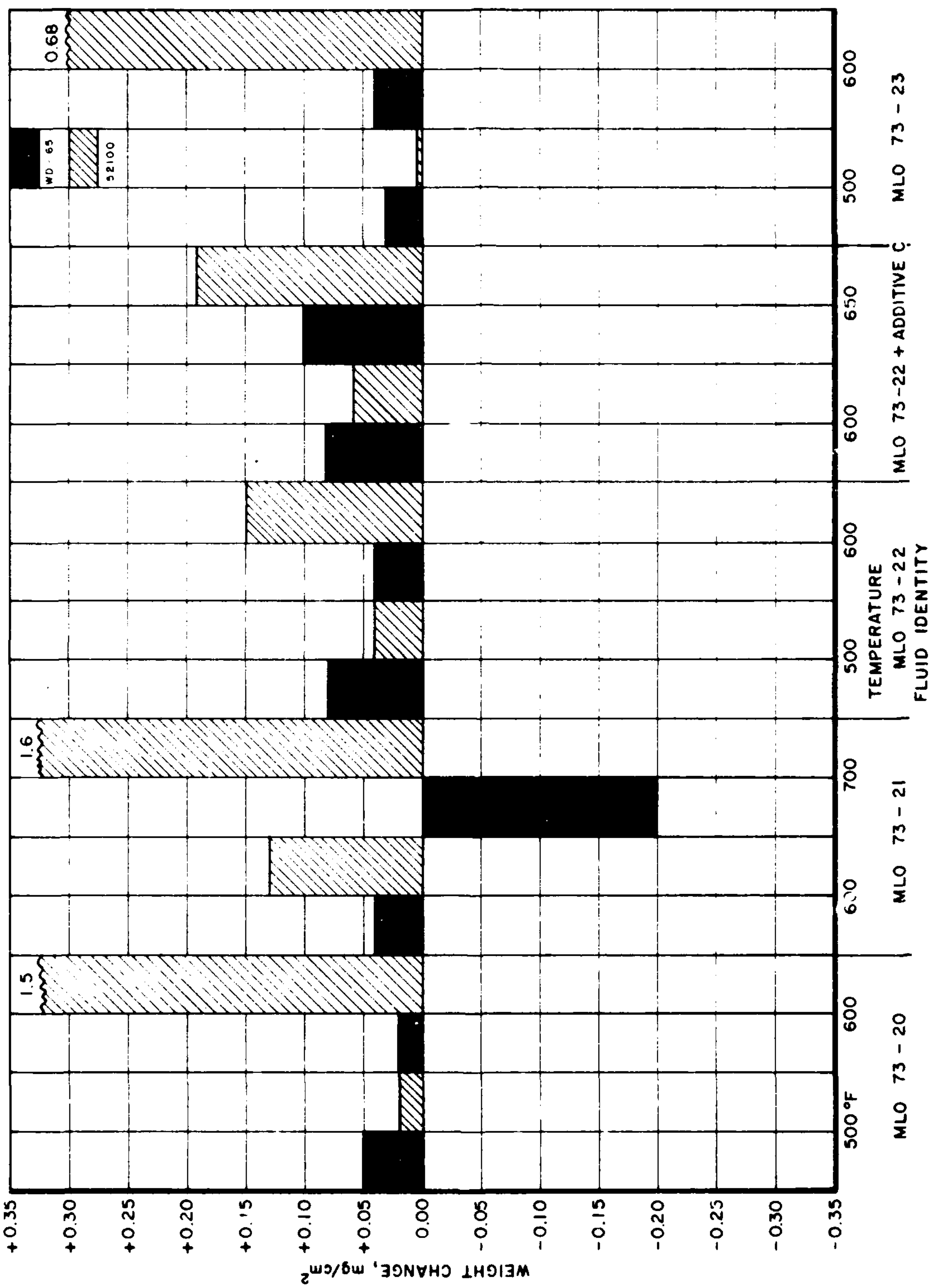


Figure 4. WD-65-52100 Corrosion Comparison

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